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PATENT SPECIFICATION

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PRINCIPAL PATENT

Sehweizerhall Acid factory, Sehweizerhall (Switzerland).

Procedure for the purification of liquid metallic chlorides.

The invention concerns a procedure for the purification of liquid chlorides of metals of the 4 group of the periodic system. These metallic chlorides are the result of well-known procedures by direct chlorination of the appropriate metals, alloys, oxides or oxide ores and are contained, because the resulting products are contaminated with the impurities, always by other volatile metallic chloride. The metal of chlorides, like ferric chloride, solid at normal temperature, are only more heavily soluble and cannot be removed in liquid chlorides for example described procedures in Swiss the patent No. 255404. On the other hand the liquid metallic chloride, like Vanadium tetrachloride, is perfectly soluble Vanadium oxide chloride, chromyl chloride, in liquid chlorides of the tetravalent metals and causes the unwanted discoloration in pure colorless chlorides of the tetravalent metals. Thus one maintains a Titanium chloride, which contains 0.7 to 0.8% Vanadinoxide chloride and Vanadium tetrachloride and to 0.1% Chromoxide chloride and is dark-yellow colored therefore for example during the chlorination from Rutilium, which always contains to 1% vanadium oxide and to 0.5% chrome oxide. During the chlorination of silica gel one maintains by small quantities (under 0.1%) light yellow colored a silicon tetrachloride to Vanadinoxide chloride. The available procedure mentioned above concerns all the purification of liquid tetravalent chlorides of these liquid, colored chlorides.

The simple distillation or even a carefully accomplished, fractionated distillation of the discolored metallic chloride do not lead to the goal, since the boiling points far be towards and colorless chlorides the too close lie. It is now well-known, the purification by means of adsorption of colored chlorides at activated charcoal to accomplish silica-gel etc. in the liquid or vapor state or the colored to convert volatile chlorides with hydrogen with higher temperature into non volatile chlorides and so from colorless chlorides to separate. Raw chlorides in the liquid were also suggested with substances such as sulfur treating soap or with metallic powders in presence from alkalis to whereby colored chlorides become by partially unknown reactions into non volatile compounds transformed, so that then a chloride, which is free from foreign metallic chloride, can be distilled off.

It was now found that the purification of chlorides of the metals of the 4. Group of the periodic system of the colored metallic chlorides more simply and cheaply in such a way to be accomplished can that one treats chlorides at increased temperature with small quantities of such metal-free organic compounds, which are carbonized by chlorides, according to which one separates the metal chlorides released from colored chlorides from that the contamination containing solid carbonization product. The separation can take place either via filtration of liquid

chloride from the solid carbonization product or via distillation of liquid chloride from the carbonization residue.

The procedure offers first of all the advantage that one approves of much cleaning agents, oils like petroleum, crude oil, paraffin oil, animal and vegetable fats and etc., use can. Secondly also such liquid substances can be used as cleaning agents, which are partly or completely in the metallic chloride soluble, as Petrol and certain mineral oil. Thus the pedantic agitating operation falls there as when using soap, sulfur and metallic powder since simmering of the Metallic chlorides attainable execution by heating is sufficient for the sufficient distribution of the cleaning agent in the Metallic chloride. Also the purification operation can be arranged continuous in simplest way by the fact that one evaporates the Metallic chloride from a distillation flask, of which, which can be cleaned, one lets small quantity of a liquid organic cleaning agent approach continuously except the Metallic chloride still another, whereby one discharges continuously or charges at least one part of blister contents and by filtration from the liquid separates the separated carbonized residue. How follows from the following examples, sees the content of colored chlorides leaves with smallest losses at colorless Metallic chloride removing practically perfectly. In this way cleaned metallic chloride leaves easily as source materials for the fabrication of perfectly white Pigment colors, as for example to titanium oxide.

Example 1:

200 g Titanous chloride, which with 2 g vanadium chloride was contaminated, were shifted with 2 g linseed oil, a half hour was cooked and distilled off then the Titan chloride. It became a water-clear product resulting with less than 0.01 weight percentage vanadium content. In the same container still sieved further exactly same loads under same additive of linseed oil were heated up and distilled off from the same, whereby a vanadium-free distillate became always resulting. After last distillation the char-like residue of all eight distillations was removed and examined from the flask; it contained all vanadium and 10 g Titan chloride, so that the loss at Titan chloride amounted to only 0.7%.

Example 2:

500 g tin tetrachloride, which with 2.5 g vanadium chloride was contaminated, were cooked with 5 g diesel oil during 2 hours and distilled. The distillate contained less than 0.01% vanadium.

Example 3:

100 g silicon chloride with 0.1% vanadium chloride were cooked and distilled for 1 hour with 0,1 g castor-oil, whereby the condensate less than 0.01% vanadium contained.

Example 4:

200 g Titan chloride with 2 g vanadium chloride were cooked with 2 g crude oil 2 hours and distilled. The distillate contained still 0.05% vanadium. To the carbonized residue now second, equal large load, was given which was likewise with 2 g vanadium chloride contaminated, under renewed additive of 2 g crude oil and distilled after two-hour cooking, whereby the distillate less than 0.01% vanadium contained.

Example 5:

200 g Titan chloride with 1.5 g vanadium chloride were cooked for fine powder anthracene with 2 g two hours and distilled then. In the condensed Titan chloride no more vanadium could be accounted for.

Example 6:

200 g Titan chloride with 2,5 g vanadium chloride were cooked 2 hours with spindle oil; after cooking the residue was filtered off and a clear, but Titan chloride with less than 0.01% vanadium resulting, darkly colored of organic decomposition products.

Example 7:

Equipment for continuous distillation with blister contents of 600 cm³ became with Titan chloride, which 1% vanadium chloride and 0.3% chromyl chloride contained, and with cylinder oil in the ratio 200:1 with a speed of 900 g Titan chloride per hour and a titanium chloride distillate with less than 0.01% vanadium and 0.005% chrome resulting. After ten hours actual working time blister contents was filtered, whereby the filtrate was returned again into the blister, which was ready then for a new operating period of ten hours.

PATENT CLAIM:

Procedure for the purification of liquid chlorides of metals of the 4 Group of the periodic system, characterized by the fact that one treats chlorides at increased temperature with small quantities of such organic, metal-free compounds, which are carbonized by chlorides, according to which one separates the Metallic chloride released from colored chlorides from that the impurities containing solid carbonization product.

SUBCLAIMS:

1. Procedure after patent claim, characterized by the fact that one separates the Metallic chloride released from colored chlorides by filtration from that the impurities containing carbonization product.
2. Procedure after patent claim, characterized by the fact that one separates the Metallic chloride released from colored chlorides by distillation from that the impurities containing carbonization product.
3. Procedure after patent claim, characterized by the fact that organic compounds are used, which are soluble in the metallic chloride.
4. Procedure after patent demand, characterized by the fact that as organic substances vegetable oils or fats are used.
5. Procedure after patent claim, characterized by the fact that as organic substances animal oils or fats are used.
6. Procedures sew patent claim, characterized by the fact, class as organic substances mineral oils to be used.
7. Procedure after patent claim, characterized by the fact that one evaporates the Metallic chloride from a distillation, of which, which can be cleaned, one lets small quantity of a carbonizable, liquid, organic cleaning agent approach continuously except the Metallic chloride still another, whereby one discharges from time to time at least one part of blister contents and from carbonized residue through filtration of the liquid separates.

Acid factory Schweizerhall.